CLASS-2

Thermal oxidation process and growth silicon oxide over the cleaned silicon wafer

- Objective
- Basic Theory of Thermal oxidation
- Growth mechanism of Thermal oxidation
- Silicon consumption during Oxidation
- Deal-Grove Model of Oxidation
- Equipment for Thermal Oxidation
- **Experimental Procedure** for Growth of Oxide layer on a cleaned Silicon wafer
- Observation & Conclusion

INTRODUCTION

- If silicon is exposed to an oxygen or air ambient. There is a chance to form a thin native oxide layer with 0.5-1nm on the surface rapidly. That oxide layer is not uniform and importantly not defect free. These two features are very vital for MOS cap. Device fabrication. That's why we have done 1% buffer HF cleaning in the previous class.

- Silicon oxide is used to isolate one device from another, to act as gate oxide in MOS structures, and to serve as a structured mask against implant of dopant atoms. That silicon oxide layers can also be produced by deposition techniques, like chemical vapour deposition, sputtering etc.

- Thermal oxidation is a complex process where a diffusion of oxidants, a chemical reaction, and a volume increase occur simultaneously to convert the silicon substrate into oxide. This process is strongly influenced by the used oxidant species, the oxidation ambient with temperature and pressure, and also the crystal orientation of the substrate. With these parameters the quality and the growth of the oxide during the manufacturing process can be controlled.

- The small dimensions and high performance of modern MOS devices require ultrathin silicon oxide layers for gate dielectrics. Apart from the exact thickness control, pure silicon oxide has some difficulties to fulfil all requirements at such thin thicknesses.

Growth of Oxide layer on a cleaned Silicon wafer

Objective:

- To perform thermal oxidation on Silicon Wafer
- To measure the thickness of the oxide layer

Theory:

➢ Thermal oxidation is the process used to grow a uniform, high quality layer of silicon dioxide (SiO_2) on the surface of a silicon substrate.

➢ The perfect interface between Si and SiO_2 is one major reason why Si is used for semiconductor devices (instead of Ge , GaAs or other materials).

➢ Silicon dioxide (SiO2) layer may be used for:

(a) to serve as a mask against implant or diffusion of dopant into silicon,

(b) to provide surface passivation ,

(c) to isolate one device from another,

(d) to act as a component in MOS structure,

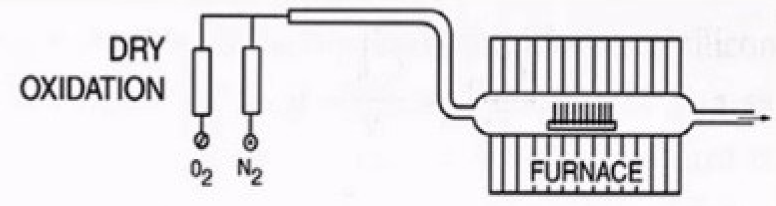
(e) to provide electrical isolation of multilevel metallization systems.

➢ At room temperature Si in air creates very thin "native oxide layer " (~1-3nm) forms on the top of the Si wafer. Native Oxide Layer is not uniform and importantly not defect free.

Growth mechanism of Thermal oxidation

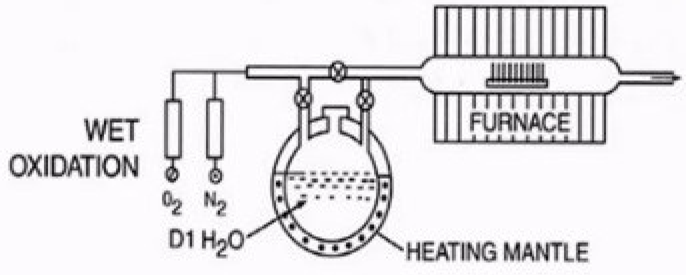
1. Dry oxidation:

Si (solid) + O_2 (gas) → SiO_2 (solid)



2. Wet oxidation :

Si (solid) + $2H_2O$ (vapor) → SiO_2 (solid) + $2H_2$ (gas)

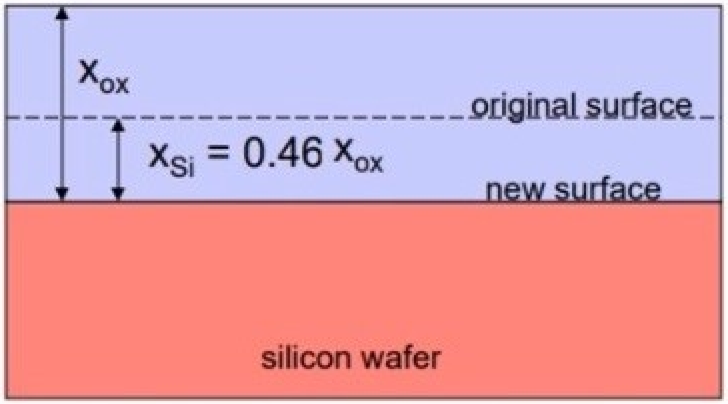


- In both cases, Si is supplied by the underlying wafer. Dry and wet oxidation need high temperature (900 - 1200°C) for growth

Parameter	Dry Oxidation	Wet Oxidation
Growth Rate	5-33 A/min	25 – 220 A/min
Thickness	< 500 nm	< 2000 nm
Quality	Excellent	Moderate

Silicon consumption during oxidation

- The mechanism of Thermal oxidation is that oxidant diffuses in oxide and reacts at interface. Silicon oxide grows at the interface and consumes bulk silicon.

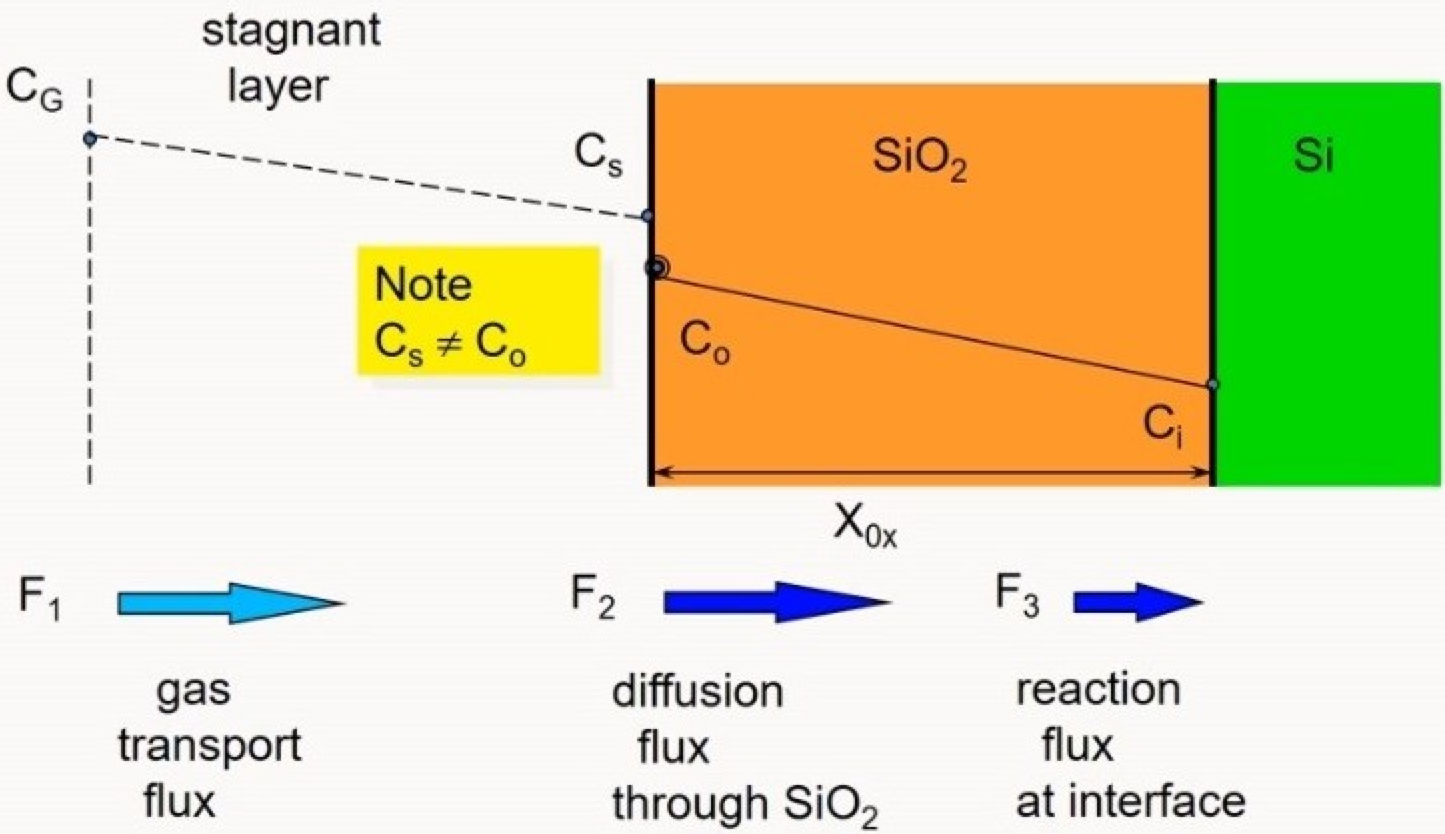


Where, X_{ox} is final oxide thickness

- SiO_2 grows 46% downward and 54% upward from the original surface

$$\frac{\text{Thickness of Si}}{\text{Thickness of } SiO_2} = \frac{\text{Molar volume (Si)}}{\text{Molar volume } (SiO_2)} = \frac{\dfrac{\text{Molecular weight (Si)}}{\text{Density (Si)}}}{\dfrac{\text{Molecular weight } (SiO_2)}{\text{Density } (SiO_2)}} = \frac{\dfrac{28.9\ \text{g/mol}}{2.33\ \text{g/cm}^3}}{\dfrac{60.08\ \text{g/mol}}{2.21\ \text{g/cm}^3}} = 0.46$$

The Deal-Grove Model of Oxidation



❖ F: oxygen flux – the number of oxygen molecules that crosses a plane of a certain area in a certain time

Deal-Grove Model of Oxidation

Oxide thickness can be measured by Deal- Grove Model

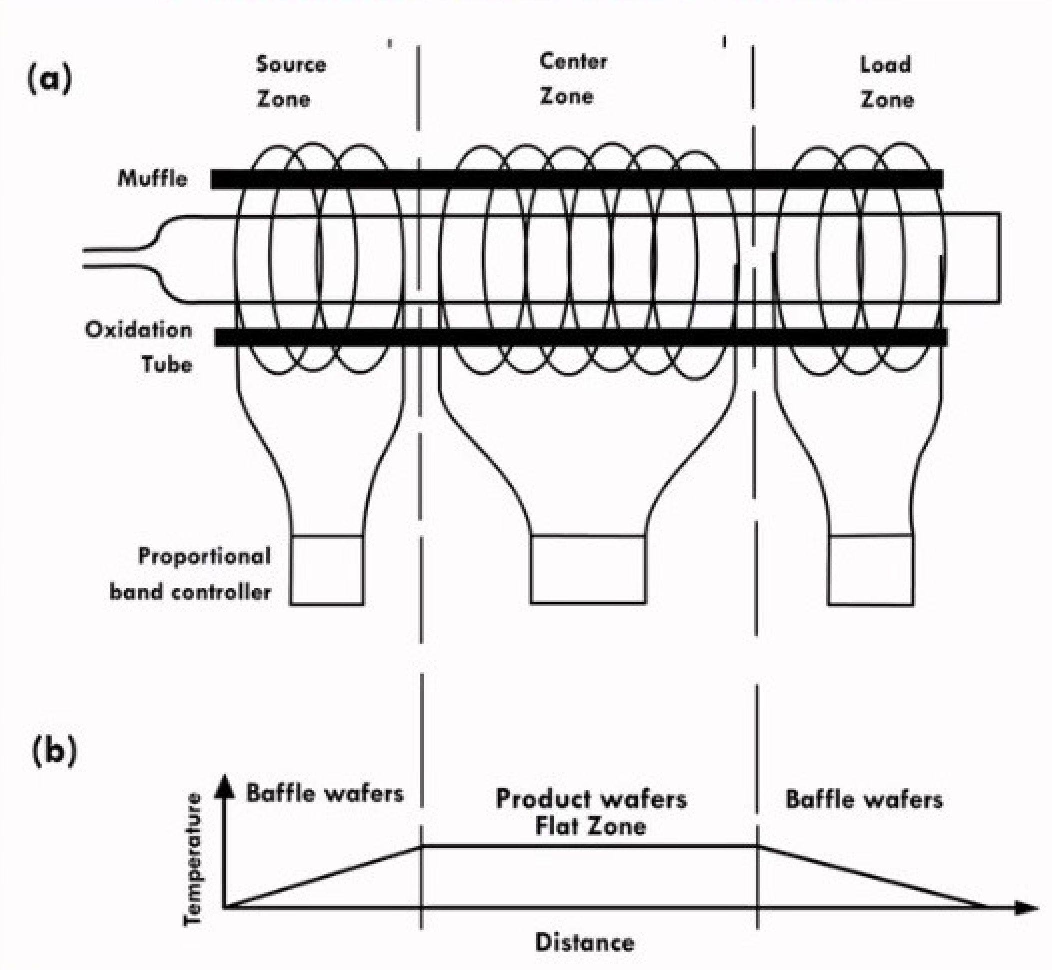
$$t_{ox} = \frac{A}{2}\left(\sqrt{1+4\frac{B(t+\tau)}{A^2}}-1\right)$$

Where, the constants A and B encapsulate the properties of the reaction and the oxide layer, respectively, and the constant τ takes into account any initial oxide thickness present. These constants are given as:

$$A = 2D_{ox}\left(\frac{1}{K_i}+\frac{1}{h_j}\right) \qquad B = \frac{2D_{ox}C_s}{N_i} \qquad \tau = \frac{t_{ox}^2 + At_{ox}}{B}$$

Where $C_s = HP_g$, with *H* being the gas solubility parameter of the Henry's law and P_g is the partial pressure of the diffusing gas. N_i denotes the oxidant molecules/unit volume needed to produce a unit volume of the o

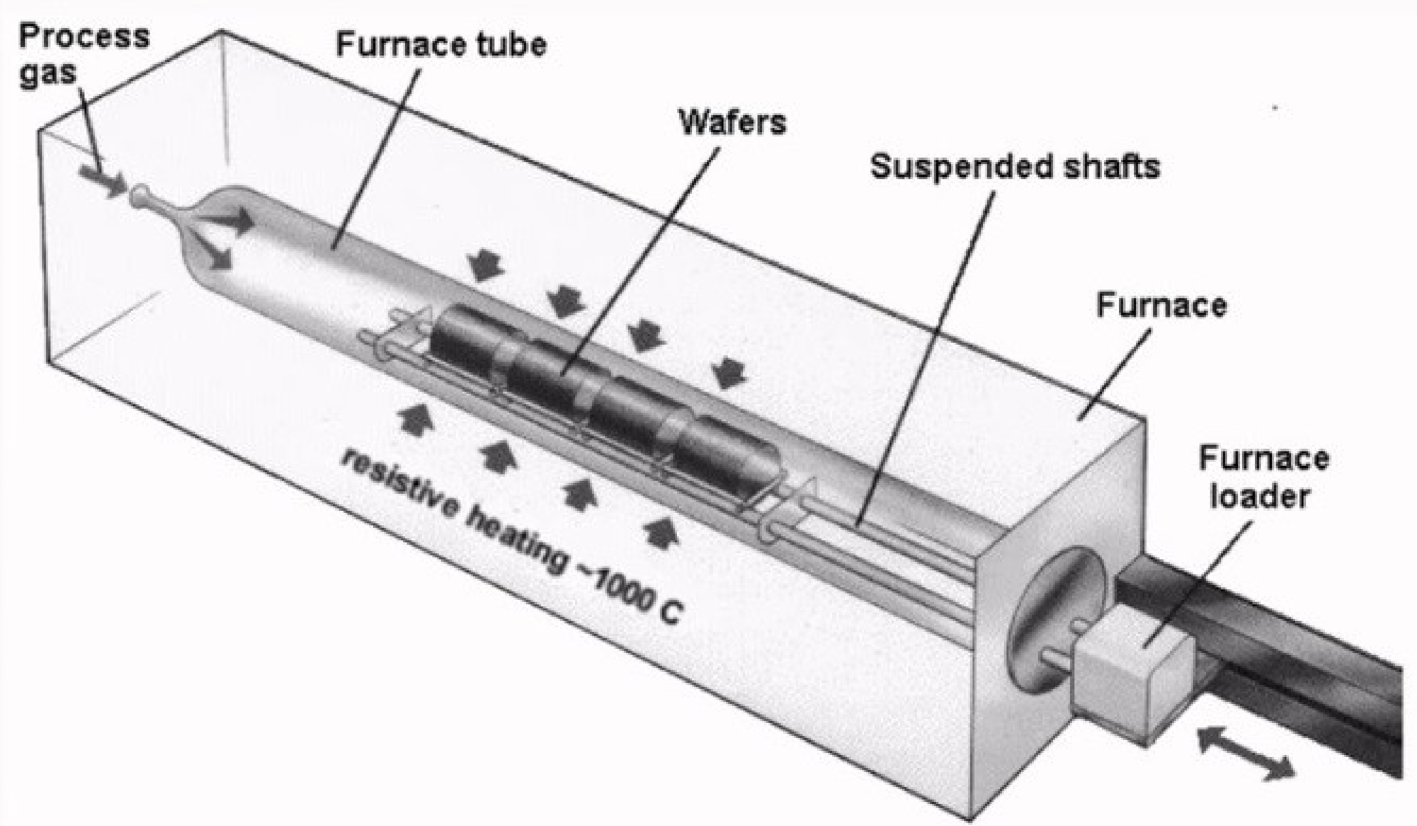
Three Zones of Horizontal Furnace



(a) horizontal diffusion furnace. (b) The furnace is typically divided into 3 zones, with the product wafers loaded in the center zone. The zones have their individual heaters and temperature controllers, to ensure uniform temperature.

Adapted from Microchip fabrication – Peter van Zant.

An internal view of the heating section of Furnace



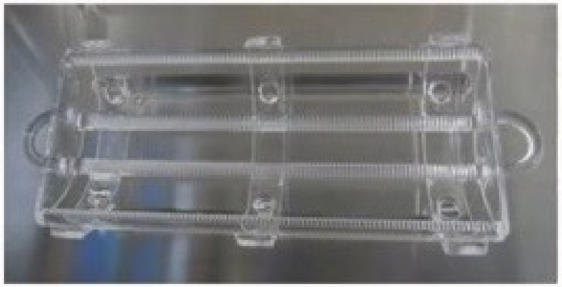
Equipment for Thermal Oxidation

A three-tube horizontal furnace with multi-zone temperature control

Vertical furnace

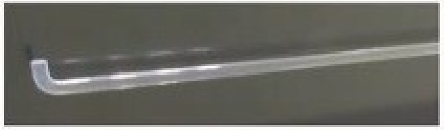
Growth of Oxide layer on a cleaned Silicon wafer ... contd.

Apparatus used for Thermal Oxidation



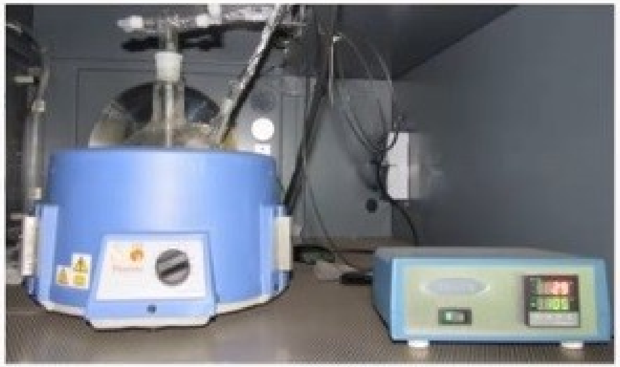
Quartz Boat

Quartz Carrier
with Quartz Boat



Quartz rod

Heat Barrier

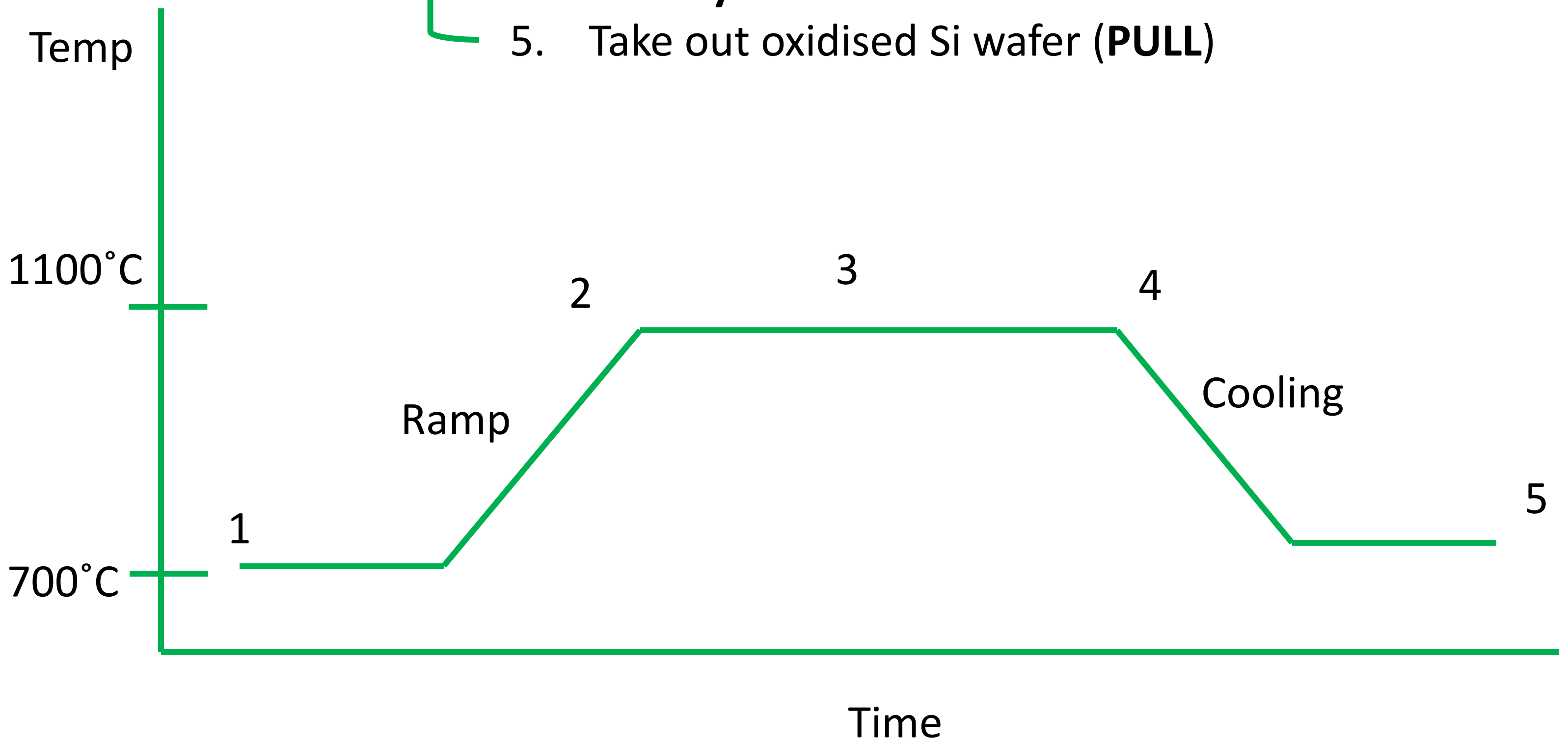


Bubbler

N2

1. Insert Si wafer+N2 (**PUSH**)
2. 1st **Dry** oxidation+N2+O2
3. 1ST **Wet** oxidation+N2+H20
4. 2ND **Dry** oxidation+N2+O2
5. Take out oxidised Si wafer (**PULL**)

N2 low



Experimental Procedure

1st Step: Turn on the Furnace and heat it up with Nitrogen gas flow of 2 slm

Before turn on the Furnace

After turn on the Furnace

N_2 Gas Flow controller

2nd Step: Take a cleaned silicon wafer from dissector and place it in the quartz boat.

3rd Step: When the Furnace temperature reaches 550^{O}C insert the quartz boat with wafer by the quartz rod inside the tube very slowly with the help of quartz carrier. Place the quartz boat at the Centre zone of the Tube.

4th Step: After placing the quartz boat, take out the quartz carriers with the help of the quartz rod. To reduce the heat loss of the Furnace put the baffle at the end of the tube and close the door of the Furnace.

Experimental Procedure ... contd

5th Step: Set temperature of the furnace at 1050°C with ramp 10°C/min.

6th Step: The entire oxidation process is divided into three steps: Dry – Wet – Dry oxidation for 10 minutes - 5 minutes - 10minutes respectively.

7th Step: At first pass pure Oxygen (O_2) gas flow is allowed inside the processing tube for 10 minutes. Flow rate of O_2 gas is kept 2 slm with the help of O_2 controller.

8th Step : After passing O_2 gas, water vapor with oxygen from bubbler is allowed inside the processing tube for 05 minutes to form thick oxide. Flow rate is 1 slm, which is controlled by N_2 and bubbler controller.

Experimental Procedure ... contd

9th Step: Then again dry oxidation is done for thin high quality oxide layer. The O_2 gas flow is allowed inside the processing tube for 10 minutes and the flow rate of O_2 gas is kept 2 slm with the help of O_2 controller.

10th Step: After the completion of the oxidation process, the temperature of the furnace is cool down to 550°C with a ramp down of 10 $^{\circ}$C/min. When it reaches at 550°C then stop the N_2 gas flow.

11th Step: When the temperature of the Furnace reduced to room temperature, then quartz boat will be taken out from the tube of Furnace with the help of quartz carrier and quartz rod. Take out the oxidized wafer from the quartz boat and keep it inside an ultra pure clean container for further processing of the oxidized wafer.

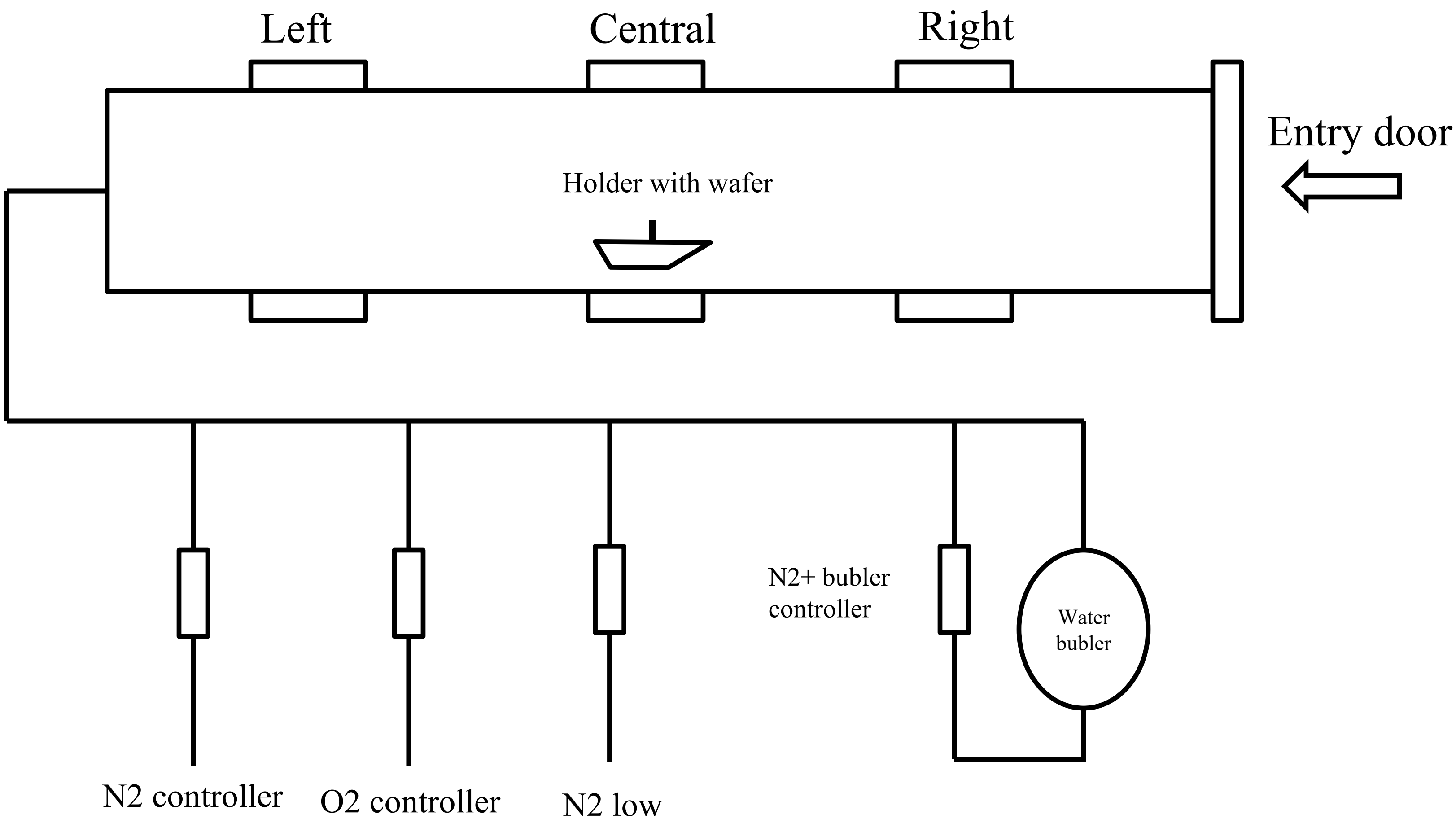
video

Thermal Oxidation Diffusion Furnace:

- Three stack & Three zone furnace
- Entire diffusion chamber is made up of quartz
- The quartz tube is segregated by three different temperature zone w.r.t the position of heating coil
- Central zone, Right zone and Left zone. the quartz boat containing cleaned silicon wafer is placed in the central zone.
- The entire tube temperature is maintained using Gaussian distribution w.r.t the three different coil temperature
- The quartz made apparatus is used to hold wafers and to closed the lid of the furnace at the entry point.
- The entire chamber is maintained inert with the continuous flow of nitrogen gas into it.

Procedure

1. Insert the quartz holder with wafer by the quartz rod, and close the lid with two opaque quartz support to resist the heat dissipation.
2. Allow temperature rising to the set value at which the oxidation process is going on, here it is 1000~1200C. The temperature ramp is 10deg/min. After process it will cool down.
3. The entire oxidation process is divided into three step: DRY-WET-DRY, time is allowed for 10min-5min-10min. In the entire process the tube is maintained inert using the constant flow of nitrogen gas which is controlled by N2 controller.
4. After temp is reached to the set value, for the 1st dry oxidation process the oxygen gas flow is allowed to inside of tube with N2 flow with the help of O2 controller
5. In the next step, 1st wet oxidation process the water vapour form bubler is allowed to of the tube with N2 flow with the help of N2 + bubler controller.
6. After that, in the 2nd dry oxidation process again the oxygen gas flow is allowed to inside of tube with N2 flow with the help of O2 controller.
7. There are two intermediate in the entire process, on that time the N2 low controller is maintained the gas flow to control the inertness of the entire tube.
8. The amount of gas flow is controlled by the controller using the unit SLM (standard litre per minute)
9. The oxide growing pattern is top to down approach. After first layer of oxide which is for dry oxidation, the next layer for wet oxidation is created below that previous layer not above.
10. Si+O2-SiO2 (dry oxidation),
    Si+2H2O-SiO2+2H2 (wet oxidation)


Left
Central
Right
Entry door
Holder with wafer
N2+ bubler
controller
Water
bubler
N2 controller
O2 controller
N2 low

Deal Grove Model

- (13) can also be written with oxide thickness as a function of time.

$$x_O = \frac{A}{2}\left\{\sqrt{1 + \frac{t+\tau}{A^2/4B}} - 1\right\}$$

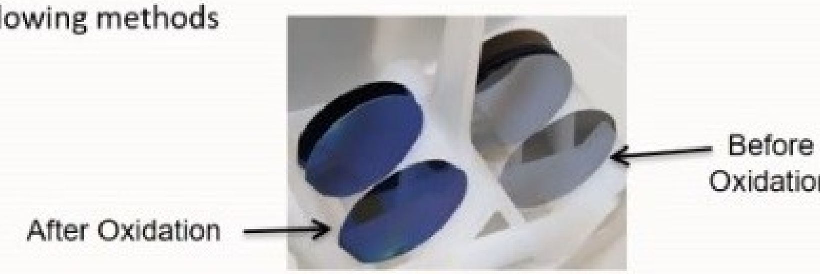
where $$\tau = \frac{x_i^2 + Ax_i}{B}$$

1. The quality of oxide is defect free and uniform thickness in dry oxidation and it is slow in growth rate. But in the wet oxidation the quality is not good but rate of grow is fast . This can helps to increment the oxide thickness.
2. The oxide rate in <111> crystal orientation is faster than the <100> orientation.
3. There is visual change in the wafer colour of presence of oxide. We are planning to grow 100 to 200nm thick silicon oxide . Depending upon the oxide thickness the incident light get diffracted and the colour changes is noticed.
4. The ellipsometry technique is used to measure oxide thickness.

Observation & Conclusion

- It is observed that after oxidation the color of the surface is changed. Before oxidation the color of the wafer would look highly **shiny and silvery**.

- Oxidation thickness can be measured by the following methods
  - Using color chart
  - Ellipsometer technique
  - CV technique
  - Surface profilometry



- From the Deal Grave model, we observe that the oxide thickness seemingly saturates with time. This is observed practically too. It is due to that fact that the growth rate would decrease as we move forward in time since O_2 molecules would face obstruction in reaching the Si molecules since due to the passage of time the oxide thickness would go higher and higher and the SiO_2 would act as a barrier. Thus the oxide thickness saturates after some point of time (i.e. thickness indirectly).

- In oxidation process we concern about dry oxidation to create high quality oxide and about wet oxidation to create thick silicon dioxide layer.

- During oxidation process 46% of silicon is consumed when compared to the total thickness of silicon dioxide layer.

- The silicon wafer was now oxidized and ready for further use.